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Method for the production of non-woven fabrics, non-woven fabrics and use thereof

The invention relates to a method for the production of non-woven fabrics, in which a lyotropic solution of cellulose carbamate in N-methylmorpholine-N-oxide (NMMNO) is spun into a plurality of filament yarns by means of extrusion through a nozzle beam containing a plurality of openings via an air gap into a regenerating bath, said filament yarns being intermingled subsequently by being subjected to a flow with gaseous medium and/or fluid. The invention likewise relates to non-woven fabrics of this type and the use thereof.

Non-wovens are textile fabrics, in the case of which the cohesion of fibres is ensured not by weaving or knitting but by interlocking and sometimes also by adhesion after intermingling of the fibres. Because of the versatile options for use and the low production costs, non-woven production still has high annual growth rates. The advantages of these non-woven materials reside in particular in the high moisture absorption, high variability of density and thickness and also the

extensive surface anisotropy, from which the numerous options for use arise, e.g. in medicine (operating sheets, bed sheets, surgical dressings, gauzes, cotton wool pads etc.), for hygiene products, as household and industrial wipes, as decorative non-woven fabrics (tablecloths, serviettes, curtains), non-woven liners in the clothing industry and also for numerous technical applications (e.g. insulating jackets in the building industry).

In principle, non-woven formation from short fibres, staple fibres or continuous filament yarns is possible. The methods of non-woven formation from filament yarns termed as "spunbonding" or as "spunlacing" have the advantage that spinning of the fibres and formation into non-wovens are effected in one process, and are the subject of this invention. A multiplicity of fibre-forming polymers can be used as starter material for the non-woven fabrics. Non-woven fabrics comprising continuous filaments are produced preferably from synthetic fibres, such as polyester, polyacrylnitrile or polypropylene. Viscose fibres are used preferably as short or staple fibres for non-woven production.

Since the viscose method, with which the largest part of cellulose regenerated fibres is still produced, involves considerable environmental damage and high investment costs, comprehensive efforts have been made already for many years to replace the viscose method with alternative methods. This applies also for the production of non-wovens comprising cellulose. Thus e.g. the so-called "Bemliese" method was developed, in which cotton linters are spun according to the cuprammonium method and processed into non-wovens (US 3,833,438). Both methods have furthermore the advantage that the non-woven products can be produced from continuous filaments in a direct method.

Another method, according to which the known "lyocell" fibre inter alia is produced, resides in precipitation of a solution of cellulose in a system comprising N-methylmorpholine-N-oxide (NMMNO) and water (US 3,767,756, DE 28 30 685), the solution being extruded into an aqueous regenerating bath via an air gap. The method is also used for the production of non-woven products (WO 00/18991, WO 98/07911).

A further known method for the production of fibres and other moulded articles comprising regenerated cellulose resides in the precipitation of a solution of cellulose carbamate (EP-A 57 105, EP-A 178 292) which is formed by conversion of cellulose with urea, with thermal cleavage of the urea into isocyanic acid and ammonia and reaction of the isocyanic acid with the OH groups of the cellulose. Cellulose carbamate is soluble in cold dilute sodium hydroxide solution and can be regenerated again into cellulose in heated sodium hydroxide solution.

Starting herefrom, it was the object of the present invention to provide a method independent of the viscose method for the production of non-woven fabrics comprising cellulose carbamate or regenerated cellulose, which fulfils the requirements with respect to low investment and production costs and also low environmental damage with good product properties. It was likewise the object of the invention to provide non-woven fabrics with superior product properties.

This object is achieved by the method having the features of claim 1 and the non-woven fabrics having the features of claims 20 and 23. The further dependent claims reveal advantageous developments. In claims 28 to 33, uses of the non-woven fabric according to the invention are indicated.

According to the invention, this object is achieved in that a lyotropic solution of cellulose carbamate in N-methylmorpholine-N-oxide (NMMNO) is spun into a plurality of filament yarns by means of

extrusion through a nozzle block containing at least 20 openings, i.e. nozzles, via an air gap into a regenerating bath, said filament yarns being intermingled subsequently by being subjected to a flow with gaseous medium and/or fluid forming the non-woven fabric.

Cellulose carbamate is soluble in NMMNO and can be shaped in a similar manner to cellulose. Relative to the conventional NMMNO method, the following advantageous differences are thereby revealed.

1. The viscosity of the solution increases greatly with increasing content of cellulose or cellulose carbamate. Too high a viscosity however impairs the spinnability of the solution. The limit of the spinnability is therefore, in the conventional cellulose solution, at a content of at most 15%. In contrast, this limit with cellulose carbamate is at approx. 30%. Solutions with a content of cellulose carbamate of 25% can still be spun without problem. The higher concentration of the solution causes lower use of solvent and hence lower expense in the reprocessing of the regenerating bath in order to recover the NMMNO and thus leads to a significant cost reduction.

2. Solutions with a cellulose carbamate content of over 20% surprisingly reveal a lyotropic behaviour, i.e. the cellulose carbamate is present in a liquid-crystalline state, as is evident from polarising microscopic pictures (pictures 1 and 2). The extremely advantageous application is produced therefore that the molecules are orientated virtually perfectly in the fibre direction during spinning as a result of the shearing in the nozzle channel, the fibres thus have an extraordinarily high orientation and hence very high strength. Strengths of 60 cN/tex and above are achievable.

3. Cellulose carbamate has a substantially higher water absorption capacity and better colouring capacity than cellulose.

Preferably a nozzle beam with at least 10,000 openings is used for the extrusion. The ratio of length to diameter (L/D ratio) of the nozzles is thereby preferably between 1 and 20.

Preferably, the width of the air gap between nozzle and regenerating bath is 5 to 250 mm, particularly preferred 10 to 150 mm.

In a preferred embodiment variant of the method, the filament yarns are guided downwards after spinning into a slot-shaped funnel, the intermingling with the gaseous medium and/or fluid being effected at the outlet of the funnel. In order to improve the intermingling of the filament yarns, it is preferred furthermore to produce this by means of a shaking movement of the funnel.

Preferably air and/or water are used as gaseous medium and/or fluid.

It is preferred furthermore that the filament yarns are laid on a conveyor belt after the intermingling. Preferably, a further intermingling of the filament yarns is achieved thereby by a shaking movement of the conveyor belt.

Preferably, the lyotropic solution is produced by swelling of the cellulose carbamate in a 50% solution of NMMNO in water and subsequent removal of the water up to a ratio of NMMNO to water between 80 : 20 and 90 : 10, particularly preferred 87 : 13.

The cellulose carbamate proportion of the lyotropic solution is thereby preferably at least 20% by weight, particularly preferred 22 to 27% by weight. The percentage details hereby relate to the entire lyotropic solution.

Preferably, the regenerating bath comprises a solution of NMMNO in water with an NMMNO proportion of 0.5 to 25% by weight, particularly preferred 5 to 15% by weight, relative to the solution of NMMNO in water.

The extrusion or spinning is effected preferably at a temperature of 80 to 110°C, particularly preferred from 85 to 95°C.

In a further advantageous embodiment variant of the method, the non-woven fabric is washed subsequent to the above-described steps, pressed and dried. The washing can thereby be effected preferably by a water jet at high pressure.

It is preferred furthermore if the cellulose carbamate is regenerated into cellulose in a regenerating bath comprising 0.3 to 1% by weight sodium hydroxide in water at a temperature of 60 to 95°C. It is made possible as a result to produce non-woven fabrics from regenerated cellulose. It is possible in a first advantageous variant to implement the regeneration between extrusion and intermingling. A further preferred variant provides that the regeneration is implemented after the intermingling.

According to the invention, a non-woven fabric is likewise provided from a random orientation of filament yarns made of cellulose carbamate. It is thereby preferred if the filament yarns have a strength of 60 cN/tex. Furthermore, a non-woven fabric of this type can be produced preferably according to the method according to one of the claims 1 to 19.

According to the invention, a non-woven fabric is also produced from a random orientation of filament yarns made of regenerated cellulose. With respect to the regeneration of the cellulose carbamate into

cellulose, reference is thereby made to claims 16 to 19. The fibres made of regenerated cellulose thereby preferably have a residual nitrogen content (residual N-content) of 0.3 to 0.5, particularly preferred from 0.1 to 1.2.

The non-woven fabric has a pore structure with a preferred porosity of 1 to 10%.

It is furthermore preferred if the non-woven fabric has a specific internal surface between 20 and 50 m^2/cm^3, measured by means of small angle x-ray scattering (SAXS).

It is particularly preferred if the non-woven fabric according to the invention can be produced from regenerated cellulose according to the method according to one of the claims 16 to 19.

The non-woven fabrics according to the invention are used preferably in medicine, in particular as operating sheets, bed sheets, surgical dressings, gauzes or cotton wool pads. Likewise the non-woven fabrics can also be used as hygiene materials or as household wipes. A further application field of the non-woven fabrics according to the invention is that of decorative non-woven fabrics, in particular tablecloths, serviettes or curtains and also non-woven liners in the clothing industry. Furthermore, the non-woven fabrics according to the invention are used as insulating jackets or reinforcing mats, e.g. as a replacement for glass fibre mats in the building industry. Because of the high strength, the non-woven fabrics according to the invention can be used, similarly to glass fibre mats, for reinforcement of plastic materials.

The subject according to the invention is intended to be explained in more detail with reference to the subsequent Figures and the subsequent example without restricting said subject to the embodiment variants described herein.

Fig. 1 shows a schematic representation of the course of the method according to the invention.

Fig. 2 shows a slot-shaped funnel according to the invention, at the outlet of which the intermingling is effected.

The basic course of the method is illustrated in Figure 1. The spinning solution 1 is hereby extruded into a regenerating bath 4 via a spinning pump 2 by means of a nozzle beam 3 which contains a multiplicity of nozzles. The spinning is thereby effected vertically from the top to the bottom via the air gap into the regenerating bath. The fibres are drawn off in the horizontal direction via deflection rollers 5. On this stretch, a first washing bath and an elongation stretch can optionally be situated. A further deflection roller 6 guides the fibre bundle subsequently downwards into a slot-shaped funnel 7, at the outlet of which the fibre bundle is subjected on both sides to a flow of air or water. The thus intermingled fibres are laid on the conveyor belt 8 situated thereunder, a further intermingling being effected by a shaking movement of the laying device or of the conveyor belt transversely relative to the running direction. The belt passes through a washing bath with a washing nozzle 9 which can also be produced by a water jet at high pressure, and hence leads to further compaction of the material in the sense of spunlacing. The conveyor belt comprises a wide-mesh net preferably made of metal, which ensures rapid discharge of the washing liquid. Subsequently, the material can be dried in corresponding drying devices. The water can however also be pressed out by a pair of rollers with which compaction of the non-woven fabric can be achieved at the same time.

Figure 2 shows the construction of a slot-shaped funnel according to the invention. The fibre can be introduced into the mentioned funnel via the fibre inlet 1. Conveyance of the fibre through the funnel is

thereby made possible by a Venturi nozzle which replaces the water jet principle. The supply of water, air or also a mixture thereof is effected through the opening 3, which, because of the Venturi profile, flows past the channel 4 such that a low pressure is produced which conveys the fibre through the channel 4. At the lower end 5 of the funnel there is situated the fibre outlet from where the filament yarns can then be conveyed further.

Example

800 g pulp with a $DP_{(cuoxam)}$ of 520 are mixed intensively in a kneader with 3,200 g of a solution, comprising 12% by weight NaOH, 30% by weight urea and 58% by weight water for 1 h at 25°C and subsequently is ripened at 23°C for 48 h to a $DP_{(cuoxam)}$ of 300. The moist alkali cellulose is kneaded for 30 min. at room temperature in a 5 l kneader with 1200 g solid crystalline urea. Subsequently the temperature of the kneader is increased to 140°C and the water which is present is drawn off. After reaching a product temperature of 140°C, the mass is kneaded further for 120 min. and subsequently discharged from the kneader. In order to obtain the pure CC, the dry crumbly mass is washed 3 times with de-ionised water at a liquor ratio of 1 : 16, is suctioned off via a frit and then dried at room temperature. This loose and crumbly product had a nitrogen content of 3.0% and a $DP_{(cuoxam)}$ of 290.

The cellulose carbamate was mixed in a kneader with a 50% aqueous NMMO solution, this solution was concentrated by withdrawing the excess water in a vacuum of 80 mbar and concentrated up to NNMO monohydrate and thereby the cellulose carbamate was dissolved. The spinning solution had a cellulose content of 25% by mass. The solution was spun on a laboratory unit with 10,000 capillaries, guided to the intermingling nozzle via a roller system, intermingled there in a water flow and laid continuously on a conveyor belt to form a non-woven,

washed and dried. The basis weight of the non-woven was 75.7 g/m^2, the dry strength 8.5 kN/m.